STUDIES ON CHEMICAL EXTRACTS OF ARECANUT AS CORROSION INHIBITORS

SYNOPSIS

OF THE Ph.D. THESIS TO BE SUBMITTED

TO

MANGALORE UNIVERSITY

FOR THE AWARD OF DEGREE OF

DOCTOR OF PHILOSOPHY

IN

CHEMISTRY

BY

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JULY 2023

Final-Synopsis of the Ph.D. Thesis to be submitted to Mangalore University

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Title of Research	**:**	**Studies on Chemical Extracts of Arecanut as Corrosion Inhibitors**
Date of Registration	**:**	**30-December-2014** **24-January-2023**
Registration Number	**:**	**MU/EXB/Ph.D./CR.04/Chem./2018-19/E.13 dated 28.05.2018** **MU/EXB/Ph.D./CR.04/Chem../2018-19/E.25. dated 24.01.2023**
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Introduction

Areca catechu L. is a palm family areca plants, perennial evergreen trees and is widely cultivated in tropical areas. in south of China, it is also widely distributed in Taiwan, Hunan, Guangdong, Hainan and Fujian. The fruit of areca catechu is popular chewable items used in traditional herbal medicine [1-5]. China is one of the world's major producers of areca nut. The main chemical constituents of areca have been identified as: alkaloids [6-9], flavonoids, tannins, terpenes, fatty acids [10-15] etc., which is also possess a wide spectrum of biological activities such as, antibacterial, anti-viral antitumor and anti-oxidation, anthelmintic action [16].

The word corrosion is as old as the earth, but it has been known by different names. Corrosion is known commonly as rust, an undesirable phenomenon which destroys the lustre and beauty of objects and shortens their life. Corrosion since ancient times has affected not only the quality of daily lives of people, but also their technical progress. [17]

Corrosion is basically the result of interaction between materials and their environment. The effects of corrosion in our daily lives. At home corrosion is readily recognised on automobile body panels, charcoal grills, outdoor furniture and metal tools. Corrosion that occurs in industrial plants, such as electrical power planta or chemical processing plans. Plants shutdown can occur as a result of corrosion. [18-20] Eight forms of wet corrosion can be identified based on appearance of the corroded metal. These are: 1) Uniform corrosion 2) Pitting Corrosion 3) Crevice corrosion 4) Galvanic corrosion 5) Erosion corrosion 6) Intergranular corrosion 7) Dealloying corrosion and 8) Environmental corrosion. [21] There are five primary methods of corrosion control: 1) material selection 2) coatings 3) inhibitors 4) cathodic protection and 5) design. [22]

Corrosion inhibitors are the chemicals that inhibits or decrease the corrosion rate in their presence at low concentration. Broadly corrosion inhibitors can be classified as organic and inorganic type corrosion inhibitors. Inorganic inhibitors can be further divided into anodic and cathodic type inhibitors. Organic inhibitors can also be classified as natural or synthetic depending upon the origin. [23-26]

Because of increase in ecological awareness and strict environmental regulations, use of traditional inhibitors is highly restricted. People started to develop eco-friendly, low cost and highly efficient plant derived inhibitors and are called as green corrosion inhibitors. Green corrosion inhibitors can be classified as natural, synthetic and designed type [27-28]. Natural green corrosion inhibitors include plant extracts, natural medicines, carbohydrates and their derivatives. Compound derived through solid phase reactions, solid supported reactions etc represents major synthetic green corrosion inhibitors. Designed green corrosion inhibitor are those whose properties including corrosion inhibition effectiveness and effect on environment can be suitably modified. Example: ionic liquids.

In the present work, it is planned to prepare green corrosion inhibitor from Areca catechu at different stages, to study their chemical composition and corrosion inhibition properties on zinc and lead metal. Three different maturation stages of Areca catechu extract were used as corrosion inhibitors they are 1) Tender areca nut 2) Mature areca nut and 3) Dry areca nut. Extraction was prepared by Soxhlet extraction method using methanol as solvent. Water extract of dry areca nut from campco industry also studied as corrosion inhibitor. Different techniques such as Fourier Transform Infrared spectroscopy (FTIR), Thermogravimetric Analysis (TGA) and Gas chromatography-Mass spectroscopy (GC-MS) were used to characterize the extracts. Such prepared extract was used to investigate their corrosion inhibitive property under varying conditions such as concentration. The results of the work have been presented in eight chapters as given below:

Chapter 1: Introduction and Literature review

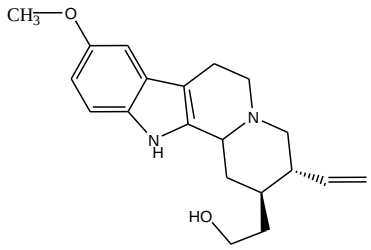
This chapter includes basic details of Areca catechu L. its chemical constituents and uses in medicine field. This section includes theory of corrosion. Economic impact on day today life, factors affecting corrosion, types of corrosion and prevention measures taken to control corrosion. A brief discussion on green corrosion inhibitor has also been covered in this chapter. Literature reviews which focus on substantive findings have been discussed to put the present work in perspective. The chapter gets concluded with the mention of the objectives and scope of the undertaken work.

Chapter 2: Materials and Methods

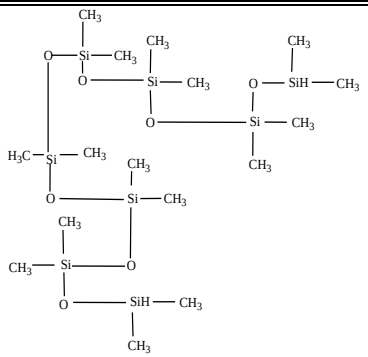
This chapter contains materials and method used in this study. This chapter is divided into two sections: The materials and chemicals used and the methodology used in this investigation. The methodology includes the procedure of collection of samples, preparation of corrosion inhibitor i.e., areca nut extracts at different maturation stages, characterization technique used as well as the experimental methods adopted for the corrosion inhibition studies.

Chapter 3: Preparation, characterization adsorption and corrosion inhibition studies of tender areca nut extract (TACN) as inhibitor on Zinc and Lead metals

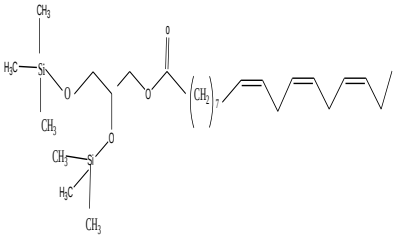
Chapter 3 explains the preparation and applicability of tender areca nut extracts as inhibitor. The characterization of extract (TACN) was performed by various techniques, such as FTIR, thermal analysis (TGA) and Gas chromatography and mass spectroscopy (GC-MS). The FTIR absorption bands of extract clearly showed the presence of some surface functional groups such as NH, OH, C=C etc., which increases the adsorption capacity of extracts. Thermal analysis data exhibited the mode of chemical decomposition of extract. Nine chemical compounds were identified from GC-MS analysis they are,



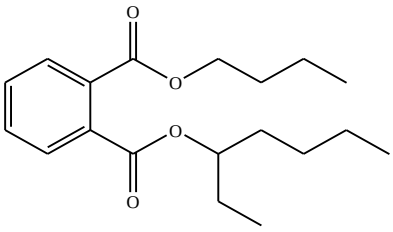
1. *2-[(2R,12bS)-3-ethenyl-9-methoxy-1,2,3,4,6,12,12b-Octahydroindolo [2,3-a] quinolizin-2-yl] ethanol*



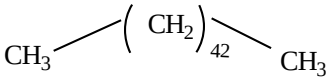
2. *1,1,3,3,5,5,7,7,9,9,11,11,13,13,15,15-Hexadecamethyloctasiloxane*



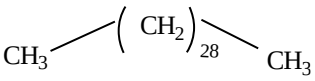
3. *2,3-bis(trimethylsilyloxy) propyl (9E,12E,15E)-Octadeca-9,12,15-triconate*



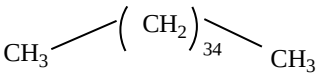
4. *Phthalic acid, butyl hept-3-yl ester*



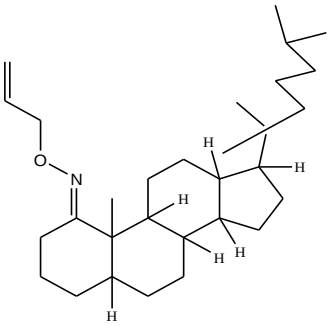
5. *Tetratetracontane*



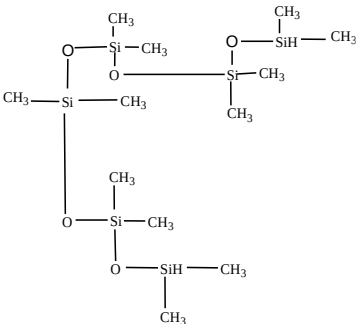
6. *Triacontane*



7. *Hexatriacontane*



8. *(E)-10,13-dimethyl-17-(6-methylheptan-2-yl)-17-tetradecahydrocyclopenta[a] phenanthrene-1-imine*



9. *1,1,3,3,5,5,7,7,9,9,11,11-Dodecamethyl hexasiloxane*

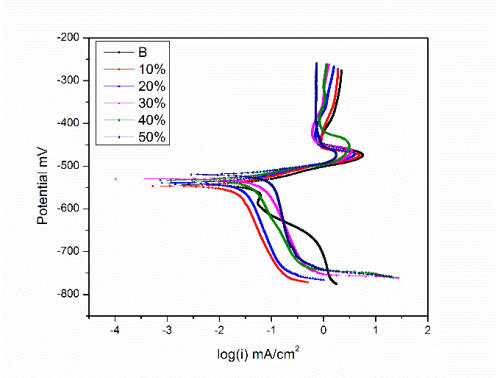


Figure.1, Polarization curves for Pb metal in 0.5M HCl solution in the absence and presence of different concentration of TACN extract

Corrosion inhibitive efficiency of TACN extract was found that corrosion rate diminishes with existence of the inhibitor concentration. Percentage of inhibition efficiency increases with increment in the concentrations of inhibitor. This is because of the extension in the measure of adsorbed molecule on the metal surface.

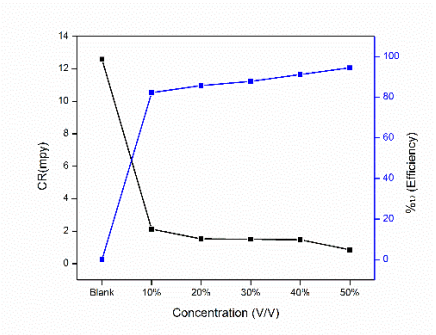
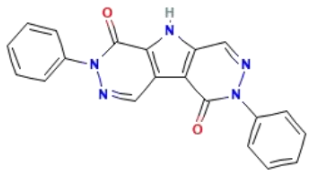


Figure.2, Variations in corrosion rate (CR (mpy)) and inhibition efficiency of metals in 0.5M HCl solution with different concentration of TACN extract.

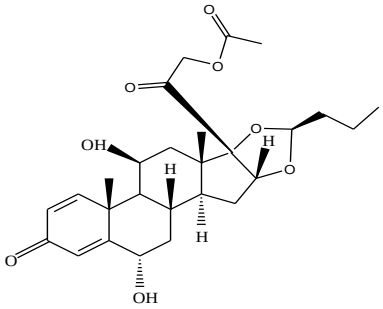
Chapter 4: Preparation, characterization adsorption and corrosion inhibition studies of Mature areca nut extract (MACN) on zinc and Lead

Chapter 4 reflects the preparation, characterization and corrosion inhibitive behavior of Mature areca nut extract (MACN). This chapter includes introduction, characterization of adsorption studies of extracts. Surface characterization was carried out by FTIR, TGA and GC-MS analysis. FTIR analysis of MACN extract. Peaks present in the IR spectra indicated the presence of few functional groups on the surface of extract and are believed to be active towards

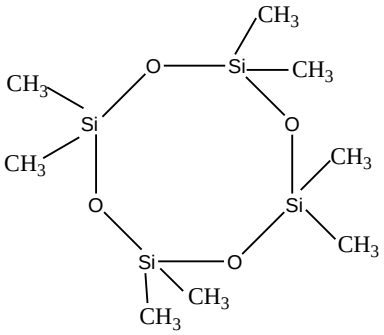
the adsorption process. Fourteen compounds were identified from GC-MS analysis and are given below,



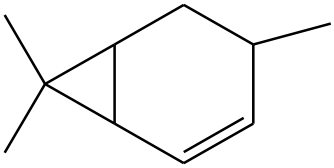
1. *4,11-diphenyl-4,5,8,11,12-pent azatricyclo [7.4.0.02,7] trideca-1(9),2(7),5,12-tetraene-3,10-dione*



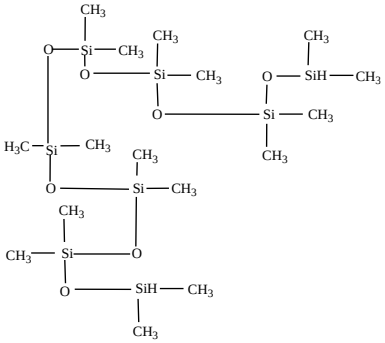
2. *(22S)-21-acetoxy-6a,11a-dihydroxy-16a,17a-propyl methylene dioxypregna-1,4-diene-3,20-dione*



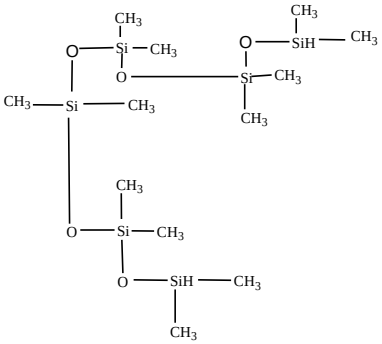
3. *2,2,4,4,6,6,8,8-Octamethyl-1,3,5,7,2,4,6,8-Tetroxatetrasilocane*



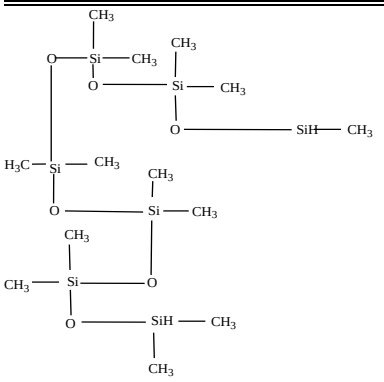
4. *4,7,7-Trimethylbicyclo [4.1.0] hept-2-ene*



5. *1,1,3,3,5,5,7,7,9.9,11,11,13,13,15,15-Hexadecamethyloctasiloxane*

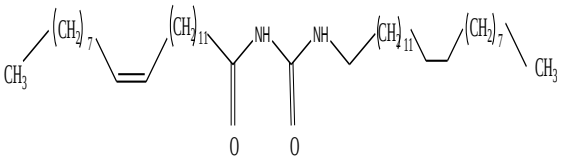


6. *1,1,3,3,5,5,7,7,9,9,11,11-Dodecamethyl hexasiloxane*

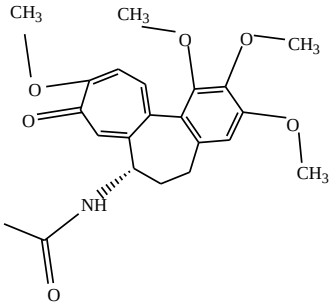


7. *1,1,3,3,5,5,7,7,9,9,11,11,13,13-Tetradeca methylheptsiloxane*

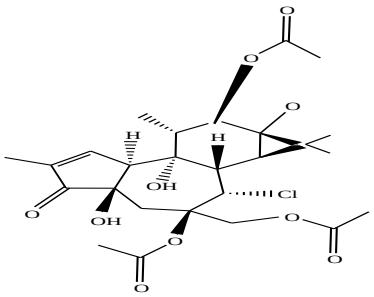
8. *(Z)-docos-13-enamide*



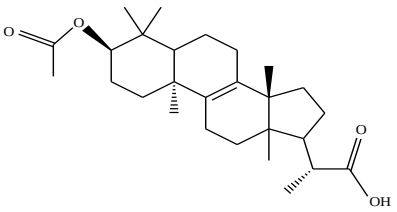
9. *(Z)-N-[[[(Z)-docos-13-enoyl] amino] Methyl] docos-13-enamide*



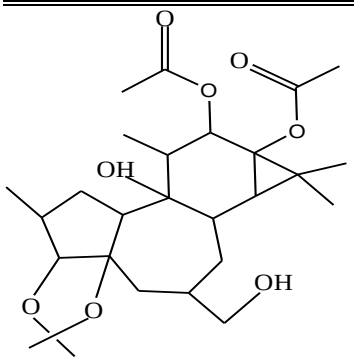
10. *N-[(7S)-1,2,3,10-tetramethoxy-9-oxo-6,7-dihydro-5H-benzo[a] heptlen-7-yl] acetamide*



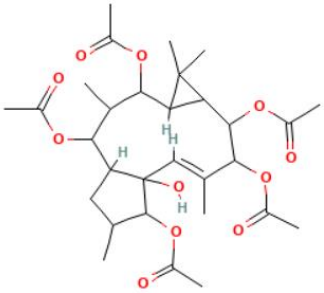
11. *[(1S,2S,6R,8R,9S,10S,11R,13S,14R,15R)-8,13,14-triacetyloxy-9-chloro-1,6-dihydroxy-4,12,12,15-tetramethyl-5-oxo-8-tetracyclo [8.5.0.02,6.011,13] pentadec-3-enyl] methyl acetate*



12. *Propanoic acid, 2-(3-acetoxy-4,4,14-trimethylandrost-8-en-17-yl)*



13. *Cyclopropa [3',4'] benz [1',2':4,5] azuleno[1,8a-d]-1,3-dioxole-5b,7,7a -triol,3a,5a,6,7,8,8a,8b,11-octahydro-10-(hydroxymethyl)-2,2,4,6,8,8-hexamethyl-, 7,7a-diacetate, [3aS-(3aà,5aà,5bà,6à, 7á,7aà,8aà,8bá,11aS*)]-*



14. *[(10Z)-4,8,9,13-tetraacetyloxy-12-hydroxy-3,6,6,10,14-pentamethyl-2-tricyclo [10.3.0.05,7] pentadec-10-enyl] acetate*

Adsorption isotherm is explained and results indicated that the equilibrium adsorption data obtained for the Extract was well described by Langmuir isotherm model.

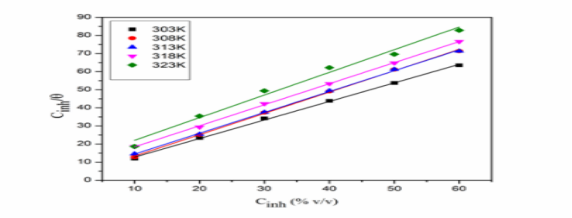


Figure.3, Langmuir plot

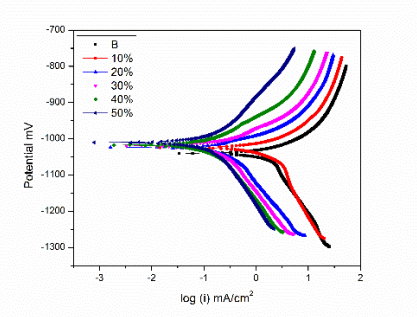


Figure.4, Polarization curves for metals in 0.5M HCl solution in the absence and presence of different concentration of MACN extract

Table.1, indicate the results obtained from polarization study of metals in 0.5M HCl solution in the absence and presence of MACN extract

Sl.No	Concentration	E_{corr} (MV)	I_{corr}(mA)	βa (mV/decade)	βc (mV/decade)	%EI
1	Blank	-1041.6	8.9519	121.720	258.32	-
2	10%	-1024.1	3.6137	97.421	236.88	59.64
3	20%	-1023.6	1.2650	89.265	185.85	85.86
4	30%	-1019.6	1.1418	82.446	180.19	87.24
5	40%	-1017.2	0.7443	67.143	170.50	91.68
6	50%	-1010.1	0.1943	52.336	107.59	95.28

Percentage efficiency and Current potential (Ecorr) was found to increase with rise in inhibitor concentration. This behaviour of the inhibitor shows that MACN extract act as effective corrosion inhibitor. The maximum shift of Ecorr is less than ± 85mV, indicating that MACN extract act as a mixed type of inhibitor.

Chapter 5: Preparation, characterization adsorption and corrosion inhibition studies of Dry areca nut extract (DACN) on Zinc and Lead metal in 0.5M HCL solution

Chapter 5 provides details on the preparation of Dry areca nut extract (DACN), the analysis of its physicochemical characteristics and the adsorption study of DACN extract. FTIR, TGA, and GC-MS techniques were used to characterize the obtained extract. FTIR analysis indicated the presence of surface functional groups like NH, OH, CH_3, C=O and C=C etc., which influence the adsorption process. Thermal analysis of the sample indicates the decomposition of the samples was attributed to evaporation of moisture content and degradation of some low molecular weight components present in the sample. Maximum weight loss at higher temperature was attributed to decomposition of phenolic acid and other chemical components.

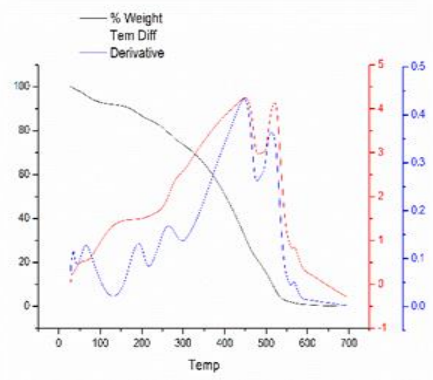
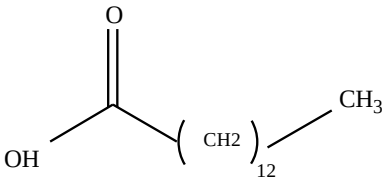


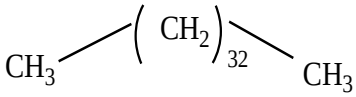
Figure.5, TGA graph of DACN extract

GC-MS studies shows the presence of fifteen different chemical compounds, five compounds were identified by comparing with the mass spectra obtained in GC-MS study, they are as follows,

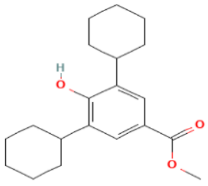


1.*Tetradecanoic acid*

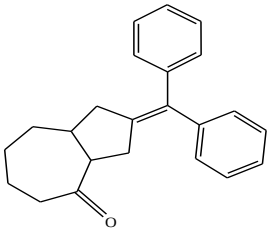
2. *(6E,10E,14E,18E)-2,6,10,15,19,23-Hexamethyl-2,6,10,18,22-Tetracosahexaene*



3. *Tetratriacontane*



4. *Methyl 3,5-dicyclohexyl-4-hydroxy benzoate*



5. *Bicyclo [5.3.0] decan-2-one,9-(diphenylmethylene)-*

Corrosion inhibition studies carried out by immersing the metal in acid solution with different inhibitor concentration.

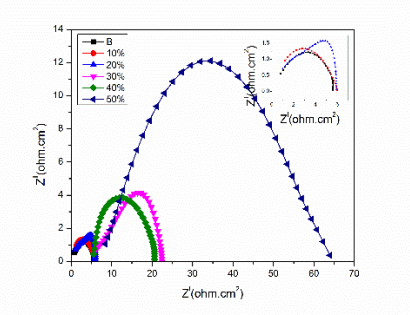


Figure.6, Nyquist Plot of metal in 0.5M HCl with and without DACN extract

Table.2, Nyquist Parameters of metal in 0.5M HCl with and without DACN extract

Sl.No	Concentration	R_s(Ω cm^2)	R_{ct} (Ω cm^2)	R_{cdl} (F/cm^2)	%EI
1	Blank	0.6791	3.864	$1.129*10^{-4}$	-
2	10%	0.6987	4.037	$3.605*10^{-5}$	4.28
3	20%	0.8724	4.628	$1.862*10^{-5}$	16.51
4	30%	9.2830	13.61	$2.779*10^{-5}$	71.61
5	40%	5.1420	16.05	$5.955*10^{-6}$	75.93
6	50%	12.09	34.36	$4.940*10^{-6}$	88.75

Increase in Rct and %EI value is because of the active components of the DACN extract hinders the corrosion rate through interfering the charge transfer process by blocking active sites of the Zn metal surface in acid media. The downward trend of Cdl value could be connected to the decrease in the local dielectric constants arising from the displacement of water molecule on the Zn surface by organic moieties present in the DACN extract.

Chapter 6: Characterization adsorption and corrosion inhibition studies of areca nut received from campco industry stored areca nut extract-1 on Zinc and Lead metal in 0.5M HCL solution

Chapter 6 provides details on the analysis of its physicochemical characteristics and the adsorption study of areca nut extract of stored areca nut extract-1. FTIR, TGA, and GC-MS techniques were used to characterize the obtained extract. FTIR analysis indicated the

presence of surface functional groups like NH, OH, C=C, NO, C-Cl, C-I etc., which influence the adsorption process. GC-MS analysis exhibit the presence of 5 different chemical species in the extract.

GC-MS study of areca nut extract received from stored areca nut extract-1 exhibit 5 different chemical compounds.

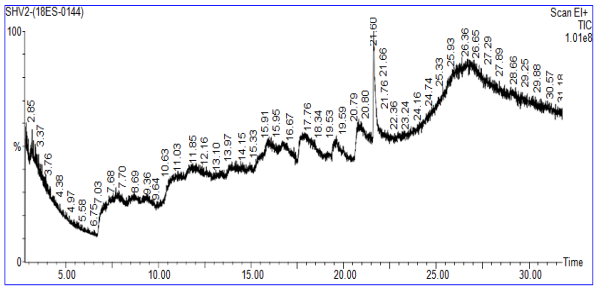


Figure.7, GC-MS spectrum of stored areca nut extract-1

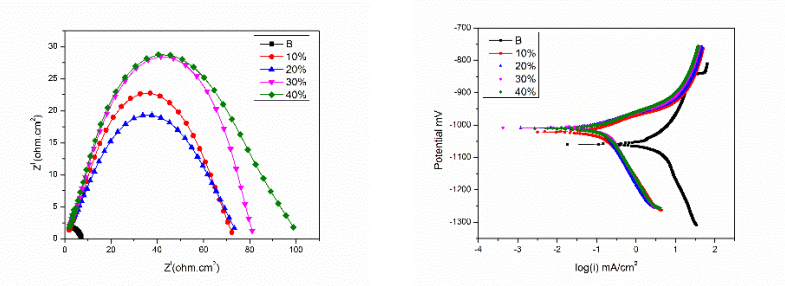


Figure.8, Nyquist and Tafel plot of the stored areca nut exract-1 with Zinc metal

It was discovered that the corrosion inhibitive effectiveness of areca nut extract received from stored areca nut extract-1 causes corrosion rate to decrease with the presence of the inhibitor concentration. With an increase in inhibitor concentration, the percentage of inhibition efficiency rises. This is brought on by an increase in the number of molecules that have been adsorbed onto the metal surface.

Chapter 7: Characterization adsorption and corrosion inhibition studies of areca nut received from campco industry, stored areca nut extract -2 on Zinc and Lead metal in 0.5M HCL solution

Chapter 7 provides details on the analysis of its physicochemical characteristics and the adsorption study of areca nut extract received from stored areca nut extract -2. FTIR, TGA, and GC-MS techniques were used to characterize the obtained extract. FTIR analysis indicated the presence of surface functional groups like NH, OH, C=C, NO, C-Cl which influence the adsorption process. GC-MS analysis exhibit the presence of 10 different chemical species in the extract. TGA analysis shows the decomposition of the extract in five different stages and most of compounds decompose in 445 to 696^{0}C temperature. Straight line obtained at the end is due to the decomposition of the metal ions present in the extract.

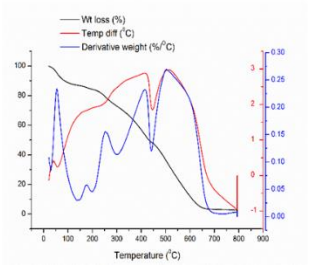


Figure.9, TGA graph of areca nut extract received from stored areca nut extract -2

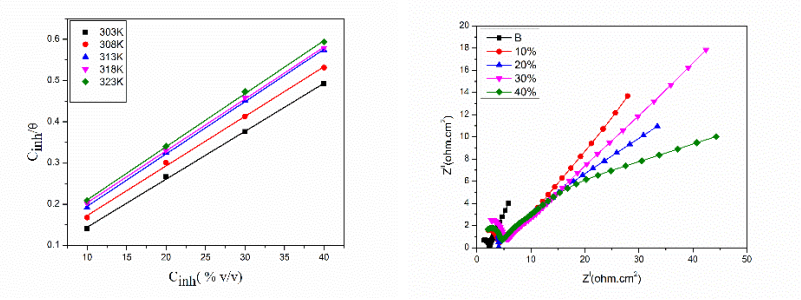


Figure.10, Langmuir and Nyquist plot for stored areca nut extract-2 respectively.

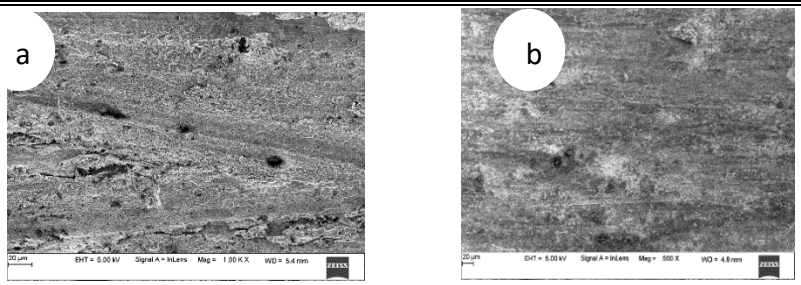


Figure.11, a and b are the SEM images of Lead metal before and after addition of stored areca nut extract-2 respectively

SEM image got from metal submerged in 0.5M HCl solutions for 3hours in the absence and presence of 40% of stored areca nut extract-2 shown in 10 (a and b). It uncovers that surface is profoundly corroded without inhibitor, is because of direct attack of aggressive acid. By correlation of SEM images at a similar amplification. It creates the impression that metal surface is free from corrosion in HCl media containing campco-2 extract as inhibitor. This is due to the development of protective film of extract on the surface. This shows that campco-2 extract restrain consumption of metal in corrosive media. Corrosion studies as shown in above graph also support the SEM studies.

Corrosion mechanism:

The gravimetric and polarization techniques, planned that areca nut extracts inhibit the corrosion process of metals (Zn and Pb) in 0.5M HCl solution, which was additionally confirmed by SEM image. Result appeared that inhibition mechanism takes place through surface assimilation method. In general, adsorption is tormented by nature of the metal and chemical structure of the inhibitor molecules. The value obtained from the above strategies will offer the knowledge concerning the mechanism of corrosion inhibition.

Areca nut extracts consists of assorted naturally occurring organic compounds. Consequently, the inhibitive action of the extract might be attributed to the adsorption of its components on the lead metal surface. The adsorption on the metal are often takes place by two possible ways. First, is inhibitor molecule could interact with the hydrogen ions and adsorption process can be takes place through electrostatic attraction between the protonated inhibitor molecules and

negatively charged metal surface due to the adsorption of the chloride ion. Second, is adsorption of the inhibitor molecules on the surface of the lead metal is due to donor-acceptor interactions. Involving reaction between heteroatoms, aromatic rings present within the Areca nut extracts and therefore the vacant orbital of the metals.

CHAPTER-8: COMPARISON OF CORROSION INHIBITION OF ALL STAGES OF ARECA CATECHU SEEDS EXTRACT ON ZINC AND LEAD METALS IN HCl MEDIA

Gravimetric and Electrochemical Studies:

In this chapter, it was mainly concentrated on the comparisons studies of Corrosion inhibition done on Zinc (Zn) and Lead (Pb) metal by two methods 1. Gravimetric method 2. electrochemical method. Corrosion inhibition study reveals that the percentage inhibition is more in case of zinc than in lead metal. Corrosion inhibition process depends on the roughness, corrosion rate increases with an increase in roughness. studies which show that larger roughness corresponds to lower pitting potential (the potential at which metastable pits start to form on the surface). We did observe that the number of pits on a smooth surface is smaller than that for a rougher surface. This implies that the formation of metastable pits on a smooth surface is more difficult than that on a rougher surface, thus corresponding to a smaller corrosion rate [6][7]. Rough surfaces had a larger interfacial area with the corrosive environment and therefore an increase in surface roughness is expected to enhance the rate of the corrosion processes.[8]

Table 8.1 shows the percentage inhibition of different types of inhibiotrs in 0.5M HCl sloution on Zn and Pb metals

Sl. No	Metals	Inhibitors of Areca seeds	%IE		
			Gravimetric study	Polarization study	EIS study
1	Zn	TACN	93.22	95.28	92.46
		MACN	87.27	94.54	89.33
		DACN	84.77	95.94	88.75
		ARC-1	87.69	80.00	88.06
		ARC-2	86.92	82.73	82.87
2	Pb	TACN	89.91	86.84	86.45
		MACN	84.12	72.54	64.33
		DACN	84.54	72.70	64.92
		ARC-1	75.28	59.69	66.74
		ARC-2	81.17	63.09	66.75

Zinc surface is rougher than lead surface, zinc oxidises more readily than lead affecting their surface topography and corrosion behaviour. Zinc corrodes relatively quickly in acidic environment than lead. In our studies we observed more corrosion inhibition value to zinc when compared to lead metal, in both gravimetric and electrochemical studies. this is because surface roughness of the zinc metal, it increases the adsorption of the inhibitor molecules, holes formed by the acid attack, traps the inhibitor molecules in irregularities formed there by reducing desorption and increasing inhibitor retention also rough surface can facilitate inhibitor diffusion allowing faster inhibition. Hence inhibition efficiency is more in zinc metal compared to lead metal.

Comparison and conclusion

Comparison between different types of prepared corrosion inhibitor extract in terms of corrosion inhibition efficiency also their chemical constituents also change as maturation occurs. This chapter also includes the summary and the conclusions drawn from this study. Result got in this shows that all the extracts hinder the rate of corrosion of zinc in 0.5M HCl arrangements. The inhibition effectiveness of the extracts increments with increment in concentration and diminishes with ascend in temperature. Polarization study uncovers that

inhibitor molecules go about as a blended sort inhibitor. Results acquired from weight reduction, polarization and EIS methods were in good understanding.

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List of Publications:

1. Sumalatha K. and J. Ishwara Bhat, Extraction, Characterization and Identification of Major Chemical Components of Areca Nut Extracts at its Different Stages, *Iranian (Iranica) journal of energy and Environment,* 10(2)136-146,2019.
2. Sumalatha K. nad Jathi Ishwara Bhat., Inhibition effect of Areca catechu Extract on Zinc in 0.5M Hydrochloric acid Solution, *Heterocyclic Letters*, 10(1)117-129,2020.

List of paper presented:

1. A paper entitled "Spectroscopic, Thermal and Chromatographic Characterization of Areca nut (Areca Catechu) Extract" (Sumalatha K, J.Ishwara Bhat) was presented in International Conference on Recent Advances in Materials Science and Biophysics (RASMB) held at Mangalore University, Mangalagangothri on January 23-25, 2018.
2. A paper entitled "Etraction, Identification and Spectroscopic Characterization of the Phytochemicals in The Seeds of *Areca catechu L*." (Sumalatha K, J.Ishwara Bhat) was presented in 37th Annual National Conference of Indian Council of Chemist, held at National Institute of Technology Karnataka (NITK), Surathkal, Mangalore- 575025 (Karnataka) on 12-14 December, 2018.
3. A paper entitled "Corrosion Inhibition Performance of Methanol Extract of Tender Areca nut (*Areca catechu*) on Lead Metal Immersed in Acid Solution" (Sumalatha K, J Ishwara Bhat) was presented in the national Conference on Recent Innovations in Medicinal and Material Chemistry (RIMMC-2019) held at Mysore University, Manasagangothri, Mysore-570006 on 8-9 March, 2019.
4. The paper entitled "An Investigation on Characterization and Anti Corrosive Properties of Aqueous Extract of Tender Areca nut in Acid Media" (Sumalatha K, J. Ishwara Bhat) was Presented in International Conference on Advanced

Functional Materials for Energy, Environment and Health Care (AFMEEHC) held at University of Mysore, Mysuru, on 18-20, March, 2019.

5. The paper entitled "Study on The Phytochemical Analysis and Corrosion Inhibition Ability of Tender Areca nut Extract as a Green Corrosion Inhibitor" (Sumalatha K, J.Ishwara Bhat) was presented in International Conference on Advances in Chemical and Materials Science, held at Mangalagangothri, Mangalore University, on 17-19 October, 2019.

6. The paper entitled "Zinc Corrosion Inhibition by Dry Areca Nut extract in Hydrochloric Acid Media" (Sumalatha K, G K Nagaraja) was presented in National Conference on Advanced Materials for Chemical and Biological Applications, held at SDM (Autonomous) College, Ujire, on 06-07 March,2024.

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